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**LONG RANGE RESEARCH LEADING TO THE DEVELOPMENT OF SUPERIOR PROPELLANTS
(DEVELOPMENT)**

Development of Deterrents for Nitroguanidine Propellants

Project No. TAL-5006B, Item r

D/A Project No.: 504-01-005

Picatinny Arsenal Memorandum Report No. MR-48 Date: 2 November 1953

F. R. Schwartz
Report by: F. R. Schwartz
Chemist (Exp)

H. A. Aaronson
H. A. Aaronson
Chemist (Exp)

Approved:

John D. Armitage
Col, Ord Corps
JOHN D. ARMITAGE
Col, Ord Corps
Chief, Technical Division
WHE *WHE* *J. V. R. K. R.*

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Agency Performing Work: Picatinny Arsenal, Dover, New Jersey
Agency Authorizing Work: Picatinny Arsenal
Ordnance Project No.: TAL-5006B, Item r
D/A Project No.: 504-01-005
DOA Priority Designation: 2A
Project Title: LONG RANGE RESEARCH LEADING TO THE DEVELOPMENT
OF SUPERIOR PROPELLANTS (DEVELOPMENT) -
Development of Deterrents for Nitroguanidine
Propellants

OBJECT

To develop deterrent coatings for small arms nitroguanidine propellants.

ABSTRACT

A first progress report is presented covering a study to date of methods for applying a deterrent coating to small arms nitroguanidine propellants. It was found that water and organic carriers for the coating agent leach out significant amounts of one or more of the major components, and may distort the grains or cause solution and recrystallization of the nitroguanidine on the surface of the grains. By the use of a new, dry-coating technique these particular difficulties have been overcome; that is, a uniform coating of organic material can be applied to the surface of the grain. However, such organic compounds were found to diffuse readily through the grain even at room temperature. The effectiveness of the initial good coating as a deterrent is thus nullified. The use of inorganic materials resulted in no diffusion but to date it has not been found possible to apply such agents so as to produce a deterrent effect. Preliminary results using organo-metallic compounds justify continued effort with this class of materials. Future work will be directed toward the use of the new coating technique to the various types of the organo-metallic class of compounds which it is hoped may be made to yield continuous, non-diffusing deterrent films.

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INTRODUCTION:

1. To date propellants containing appreciable amounts of nitroguanidine have been used only in large caliber artillery weapons. In the course of some studies of nitroguanidine propellants at this Arsenal (Ref A and B), it was suggested that such compositions would also be desirable for use in small caliber automatic weapons because the lower burning temperature associated with such compositions could be expected to result in increased barrel life.

2. At about the same time, it was learned that other installations (Ref C and D) had already started experimental work directed to the above end and had reported unsuccessful efforts to apply the necessary deterrent coatings whereby the control of the burning rate of small arms propellants is obtained. This report gives the results of preliminary work done at Picatinny on the problem of coating small arms propellants containing appreciable amounts of nitroguanidine.

DISCUSSION:

3. The M15 and M17 propellants used in the experimental work have the following nominal compositions:

<u>Component, %</u>	<u>M15</u>	<u>M17</u>
NC (13.15%N)	20.0	22.0
Nitroglycerin	19.0	21.5
Nitroguanidine	54.7	54.7
Ethyl centralite	6.0	1.5
Cryolite	0.3	0.3

The propellants thus consist of a plastic matrix of about 40% NC / NG, mechanically mixed with nitroguanidine. The usual technique of coating small arms IMR propellant, which contain neither NG nor nitroguanidine, involves the use of water in which the grains are tumbled together with the coating agent at a temperature above its melting point. In this manner, the coating agent is liquefied and can be uniformly distributed and absorbed by the grains. However, since nitroguanidine is appreciably soluble in water at elevated temperatures, and since nitroglycerin is soluble in or dissolves many organic compounds, some modification of the usual coating technique would be necessary.

4. In order to obtain some basic information regarding the types of solvents which might be used as possible carriers for coating agents, a series of solvents of varying polarity was tried. The solvents were stirred with some propellant grains to determine the extent of leaching of soluble components and possible structural changes in the grain. The results of this series of tests are given in Table I. It was found that all the solvents tried leached out a significant amount of the composition.

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DISCUSSION: (cont)

Active solvents, such as the lower alcohols, ketones and esters, softened the nitrocellulose binder and caused distortion or completely destroyed the grains. Water, higher alcohols and the aliphatic hydrocarbons were found to have the least effect at room temperature. Water, and 1/1n-propanol/water mixture extracted approximately 3% and 8.0% respectively of the nitroguanidine; while hexane, cyclohexane and Solvesso #2 (a petroleum solvent boiling at 140 - 180°C) leached out about 1.6, 1.2 and 6.4% nitroglycerin respectively. Based on these results, coating of the grains in a water slurry, even at room temperature, is considered to leach out too much nitroguanidine from the basic composition. In addition, some of the extracted nitroguanidine was found to have recrystallized in long needles on the surface of the grains.

5. Water emulsions of several polymers were tried, but proved to be just as impracticable as the other carriers. The grains were distorted, excessive clustering resulted, and the coatings obtained were non-uniform. The polymers tried included polystyrene, rubber, vinyl acetate, a copolymer of vinyl acetate and vinyl chloride, cellulose acetate, methyl methacrylate, and polyvinylacetate. Several emulsions with higher melting waxes, including paraffin, carnauba, and candelilla were also tried without any success. Coatings from solvents other than water presented problems similar to the above. As previously noted, in paragraph 4, active solvents distorted the grains, leached out nitroglycerin and served to soften or weaken the nitrocellulose binder. Inactive solvents, such as the aliphatic hydrocarbons, had the shortcoming that few coating agents were sufficiently soluble, and those that were soluble caused considerable clustering of the grains.

6. Based on the above experience, it was decided to try to apply coatings without the aid of a solvent or carrier, i.e., to use a dry-coating technique by which the coating agent could be made to adhere to the surface of the grain. It was reasoned that the temperature at which the grains should be coated would have to be below the melting point of the agent to avoid bonding of the grains to each other. Also, because of the adverse effect of high temperatures, particularly on the stability of the nitroglycerin, it was decided to use a maximum temperature of 70°C to effect the coating. As noted in paragraph 3, the propellants involved, consist of approximately 55% nitroguanidine dispersed in a matrix of a 1.1 plasticized mixture of nitrocellulose and nitroglycerin. This last compound is not a particularly good solvent for nitrocellulose (Ref E). The high ratio of nitroglycerin to nitrocellulose would be expected to leave some of the former uncombined with the latter and therefore comparatively mobile. This mobility of the nitroglycerin which would be appreciably increased at more elevated temperatures would result in some migration to the surface. Since nitroglycerin is an excellent solvent for many organic compounds, it would dissolve some of the coating agent. As the resultant solution would have a lower fusion temperature than the coating agent alone, it might be possible to effect the desired coating appreciably below the temperature of the melting point of the additive.

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DISCUSSION: (cont)

7. To test the above idea, a laboratory size sweetie barrel was designed and built which would permit coating experiments to be made with as little as 20 grams or as much as 500 grams of the propellant. A series of solid organic compounds were tried without any immediate consideration of their possible deterrent effect. These compounds, together with some details of their melting points, coating conditions and the nature of the resultant coatings are listed in Tables II and III. In general, the technique was successful in coating the grains, thus strengthening the probable validity of the concepts expressed in the previous paragraph. It was found that if the initial coating temperature was too close to the melting point of the compound, it either stuck to the barrel or caused excessive clustering of the grains. The more active the coating agent as a gelatinizer for nitro-cellulose the greater was the tendency for the compound to act as an inter-granular binder with resultant clustering. Coatings were applied in most cases at temperatures several degrees below the melting point of the coating agent and in amounts ranging from 1% to 6% of the weight of the propellant.

8. The dry coating technique showed considerable promise as a means of coating the grains with organic materials. There was no dimensional distortion as in the case of active solvents. Also, the comparatively low temperatures of application and lack of carriers presumably prevented any significant changes in composition due to losses in nitroglycerin. As much as 6% of the coating agents could usually be applied in a relatively short time with a negligible amount of clustering. The coating agents not only coated the grain but also penetrated it to some extent. It was thought that this would enhance the deterrent effect. In these coating experiments the central perforation usually contained comparatively little of the coating material. This too is considered desirable as it would increase the deterrent effect by allowing the grain to burn initially more rapidly from the inside out rather than from the outside in.

9. In spite of the promising results obtained with the dry coating technique as pointed out above, the grains coated with organic compounds soon showed one grave undesirable characteristic. The coating agents did not remain on and near the surface of the grains but continued to diffuse even when held at room temperature. This was readily apparent from an examination of grains coated with highly colored materials, e.g., 2-nitro-diphenylamine. Photographs of cross and longitudinal sections taken at intervals clearly show this diffusion. See photograph M-43965 attached. The dark central perforation on the longitudinal sections is primarily an optical effect as can be seen by comparison with the cross-section of a similarly treated grain. Photograph a was taken immediately after coating. The other two, viz. b and c, are pictures of the same grain as a, showing the extent of diffusion after 6 and 24 hours respectively at ambient temperature. Observations on the diffusional behavior of the various coatings are noted

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DISCUSSION: (cont)

under "Remarks" in Table III. This diffusion may be explained on the assumption that the structure of the grain is sufficiently porous to permit the migration of nitroglycerin to the surface where it can combine with the coating agent, but subsequently by dynamic interchange, migrates back carrying some of the organic compound in solution. The rate of diffusion depended on the nature and amount of the coating and on the temperature at which the coated grains were held.

10. It was suggested that a preliminary coating of the grains with colloidal carbon blacks, because of their extreme fineness and absorptive properties, might act as mechanical barriers to prevent the interdiffusion of nitroglycerin and the subsequent coating on the carbon black. Batches of nitroguanidine propellant were tumbled with four different types of commercially available carbon blacks. It was found that these remained on the surface without penetrating into the grain. The carbon coated grains were then treated with 2-nitrodiphenylamine by the dry-coating technique. After standing overnight at ambient temperature, the organic coating had diffused into the grain. See Table III, Experiment Nos. 24 through 27. Thus, the carbon layers were not effective in sealing off the surface of the grain.

11. Attention was then directed to the study of inorganic solids as possible coating agents. A number of materials were tried. It was found that these could readily be applied by the dry-coating technique in amounts up to at least 2% (the maximum tried). The results are included in Table III. These 2% coatings appeared to be continuous and showed no penetration into the grains when examined under the microscope. Very little coating material was observed in the central perforations of the grains. Closed bomb tests were made on grains coated with boron nitride, silica, aluminum and molybdenum sulfide. These tests showed that on burning the progressivity of the coated grains was about the same as that of the uncoated propellant. Also, the grains coated with these inorganic materials showed decreased ignitibility as indicated by the increased time of delay required for ignition. Because of the lack of deterrent action, it is believed that the coatings were discontinuous in spite of the apparent observed continuity and therefore did not significantly alter the burning characteristics of the propellant.

12. To obtain a uniform coating with an inorganic compound, the expedient was tried of adding some organic solid as a possible carrier for the inorganic material. The organic compound was first blended with the inorganic compound and the mixture applied by means of the dry-coating technique. This procedure was tried with several inorganic substances using 2-nitrodiphenylamine as the carrier. The latter compound diffused into the grain leaving the inorganic material behind as a coating on the surface. Closed bomb tests on the propellants so treated showed no

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DISCUSSION: (cont)

significant improvement in burning characteristics.

13. Since organic compounds gave excellent coatings but diffused and inorganic materials gave poor coatings but did not diffuse, it was thought that a combination of the desirable properties of each might be found in the intermediate organo-metallic compounds. The compounds tried were the salts of available fatty acids. Grains were coated with the stearates of copper, strontium, calcium and aluminum, and with copper resinate. In each case the coating was discontinuous and powdery, so that it readily rubbed off. See Table III, experiment numbers 39 through 48. Unlike the organic compounds which are appreciably soluble in the small amount of free nitroglycerin present at the surface of the grain, permitting coating at temperatures below the melting point of the organic material, the organo-metallic compounds were not soluble in nitroglycerin. Thus, their melting points were not lowered so as to permit a continuous coating to be formed even at a temperature of 90°C, 20°C above the originally mentioned 70°C coating temperature. Attempts were made to improve these poor coatings by the use of a carrier which would at least be a partial solvent for the coating-agent. Disregarding diffusion of the carrier for the time being, it was thought that such an agent would make possible a more continuous and uniform coating. Dicyclohexylphthalate was such a partial solvent and was tried with copper stearate as well as copper resinate, see Table III, Experiments 52 through 58. The copper salts were of particular interest because their green color permitted good visual examination of the extent and condition of the coatings. These appeared to be continuous although non-uniform. See photograph M-43965/1 attached. The coated grains showed pronounced surface tackiness which caused them to stick together on standing. However, in view of the apparently improved surface coating obtained by the above technique and the absence of diffusion shown by the organo-metallic coatings even after storage at ambient temperature for three months, it is considered desirable to continue the study of the use of such compounds.

14. The work to date has resulted in a process whereby diverse materials can be readily applied as coatings on small arms nitroguanidine propellants, i.e., the dry coating process and has indicated some additional lines of approach by this new technique. Among these may be included:

a. deposition of an organic polymer on and possibly near the surface of the grain; the polymer either to be deposited as such or formed from the monomer during the coating operation.

b. coating with organo-metallic compounds, e.g. metallic salts of organic acids, chelates, coordination complexes etc. It is hoped that from these, compounds may be selected which will yield effective deterrent coatings without diffusing. If this cannot be done by direct coating then various carriers will be tried.

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EXPERIMENTAL PROCEDURES:

15. To determine the effect on the grains of various solvents that might be used as carriers for the coating agents, a series of liquids of different polarities were selected. A mixture of 5 grams (approximately 147 individual grains) of 90 mm M17 propellant were agitated in a 125 ml Erlenmeyer flask with 25 ml of liquid. After two hours the grains were separated from the liquid and dried to determine the loss in weight. The liquid was evaporated to dryness and weighed. A check on the extracted matter was thus obtained.

16. A small sweetie barrel was built for laboratory experiments. It was designed to duplicate plant operations on a laboratory scale. The usual procedure was to tumble the grains of caliber .50 propellant together with the desired amount of coating agent for about 5 to 10 minutes at room temperature. This served to distribute an initial fine film of the coating material uniformly over the surface of the grains. The barrel was then partly immersed in a water bath whose temperature was maintained and controlled by the injection of live steam. Because of the size of the bath, the temperature could readily be held at $\pm 1^{\circ}\text{C}$ of that desired. The temperature of the bath was then raised until the coating agent was adsorbed onto the surface of the grains. Details of the procedure were varied in accordance with the nature of the coating material. See Tables II and III for the initial and final temperatures used. In the case of the organic compounds, the coating temperature was usually several degrees below the melting point of the compound. The coating temperature was maintained for a period of 15 to 20 minutes. At no time was the temperature permitted to rise to the melting point of the coating compound as this resulted in an agglomeration of the grains. The barrel was then removed from the bath and allowed to cool in the air while the grains were tumbled. A stream of compressed air was directed into the barrel to hasten the cooling operation. After reaching room temperature, the contents of the barrel were transferred to a 16 mesh sieve under which were placed a 12 mesh sieve and a base pan. The sieves were shaken and the clustered grains and fines separated from the normal grains retained on the 16 mesh sieve. In each case, the amount of clustering, i.e., the portion held on the 12 mesh sieve was determined.

17. Closed bomb tests were made on a number of the coated samples in comparison with the uncoated propellant to determine whether the coating had a deterrent effect. These tests were made in accordance with the procedure standard at the Arsenal (Ref B).

18. After coating, the grains were examined under a Bausch and Lomb binocular microscope at a magnification of 40 diameters. The grains were sectioned for examination by means of a sharp blade to minimize stripping of the coating agent.

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REFERENCES:

- A. 1st Indorsement dated May 5, 1952, O.O. 471.5/65(c), ORDBB 471.5/3309-786.
- B. Letter to E.I. duPont dated 16 August 1951. Contract NOrd 9691.
- C. Contract NOrd 9691 "Coating Nitroguanidine Propellants".
- D. Contract DAI-23-072-501-ORD-(P)-7, "Development of a Ball Powder Using the M17 Formulation".
- E. PA Technical Report No. 1786, "The Kinetics of Solution of Cellulose Nitrates in Explosive Plasticizers".

INCLOSURES:

Tables I, II, III
Photograph M-43965
Photograph M-43965/1

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TABLE I

LEACHING EFFECT OF VARIOUS SOLVENTS ON 90 mm M-15 PROPELLANT

(Extraction Time 2 Hours by Mechanical Shaking at Ambient Temperature)

Expt. No.	Solvent (50 ml.)	Wt. of Sample (gms)	Wt. of Leached prod. (gms)	% Leached	Remarks
1	water	4.9789	.1516	3.04	Product chiefly nitroguanidine
2	methanol	4.9657	-	-	Grains completely disintegrated
3	ethanol	5.0108	.6394	12.76	
4	n-propanol	5.0175	.3494	6.96	
5	ethyl methyl ketone	4.9867	-	-	Grains completely disintegrated
6	nitroethane	5.0027	-	-	Grains completely disintegrated
7	trichloroethylene	5.0008	.3664	7.33	
8	benzene	5.0262	1.1549	22.98	
9	xylene	4.9960	.3849	7.70	
10	hexane	5.0292	.0766	1.55	Extract chiefly nitroglycerin
11	cyclohexane	5.0142	.0584	1.16	Extract chiefly nitroglycerin
12	isopropyl ether	5.0336	.3464	6.88	
13	kerosene	5.0163	.0684	1.36	Extract chiefly nitroglycerin
14	turpentine	4.9877	.1068	2.14	Extract chiefly nitroglycerin
15	gasoline	5.0034	.1233	2.46	Extract chiefly nitroglycerin
16	Solvesso #2	5.0732	.3238	6.38	
17	n-propanol/water:50/50	4.9825	.4174	8.38	Extract essentially nitroguanidine
18	carbon tetrachloride	5.0059	.1522	3.04	
19	n-butyl alcohol	5.0104	.3392	6.77	
20	ethylene dichloride	5.0172	1.1563	23.05	
21	toluene	5.0547	.5018	9.93	

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TABLE II

INITIAL COATING EXPERIMENTS IN 70 mm 4-15 MIFOMAXISINE POLYMERIZING GRAINS

Expt. No.	Coating Agent	M.P. °C	Coating Temp. °C	Coating Time, min.	Remarks
1	methyl β-naphthyl ketone	52-3	53	20	Material gave poor coating - grains stuck to barrel - some clustering of grains.
2	formyl diphenylamine	73-4	54	15	Grains clustered at coating temperature - coating appeared uniform - grains very tacky at room temperature.
3	methyl β-naphthyl ketone	52-3	48	5	Grains coated but completely softened by the coating agent.
4	ethyl oxanilate	65-6	48	10	Grains completely coated and softened - some clustering.
5	diphenyl nitrosamine	66-7	58	5	Grains coated but coating agent readily peeled off.
6	octadecylphthalide	71-2	58	15	Grains coated at elevated temperature - coating became powdery at room temperature.
7	diphenylethylendiamine	65	56	5	Grains coated and excessive amount of clustering.
8	phenyl α-naphthylamine	60	54	7	Grains coated and excessive amount of clustering.
10	di-o-tolyl ethylene diamine	60-6	60	10	Good coating - grains softened.
11	diphenoxydiethylether	66-7	67	7	Grains coated - clustering.
12	diethyl centralite	72-3	50	10	Excessive clustering.
13	2-nitrodiphenylamine*	75-6	60	7	Material coated very well - grains softened.
14	diphenylaniline	70	66	5	Grains coated and excessive amount of clustering.
15	β-naphthylmethyl ether	72	65	5	Grains coated and slight amount of clustering.
16	p-ethoxydiphenyl	71-2	67	5	Grains very tacky.
17	dimethylacetone	73-4	55	5	Grains coated and slight amount of clustering.
18	benzyl α-naphthyl ether	76	71	7	Grains coated very well - grains softened.
19	o-nitroaniline	72	60	5	Coating agent apparently sublimed.
20	benzofuran	72	60	5	Grains very tacky - clustered.
21	diphenylformaldehyde	72	60	5	Grains stuck to barrel - clustered.
22	methoxydiphenyl	84-5	60	5	Grains stuck to barrel - grains softened.
23	benzyl phenyl nitrosamine	57	58	5	Very little clustering - grains softened.
24	nitrodiphenylamine	58-9	58	7	Grains stuck to barrel.
25	benzoylacetophenone	57-8	58	5	Grains stuck to barrel - uneven coating.
26	diphenylamine	51-2	58	5	Grains tacky and clustered.
27	isopropoxy diphenylamine	86-7	58	30	Very little clustering - grains softened.
28	di-o-tolyl ethylene diamine	75	69	7	Grains stuck to sides of barrel - some clustering.
29	2,4-dinitroanisole	84-5	69	15	No clustering or tackiness, perforation caused by coating agent.
30	tetramethyldiaminodiphenylmethane	80	69	20	Coating agent stuck to barrel - no tackiness or clustering.
31	tribenzylamine	92	70	55	Coating agent balled-up - uneven coating - grains clustered.
32	di-p-tolyl ketone	95	70	20	Coating agent balled-up to barrel.
33	triphenylmethane	96-7	70	50	Grains coated - no tackiness or clustering.
35	1,2-bis(2-biphenyloxy) ethane	-	70	40	Grains clustered together.
36	ethyl centralite	72-3	70	5	

* Grains coated with 1,3 and 5,5-dinitrodiphenylamine submitted for identifiability tests.

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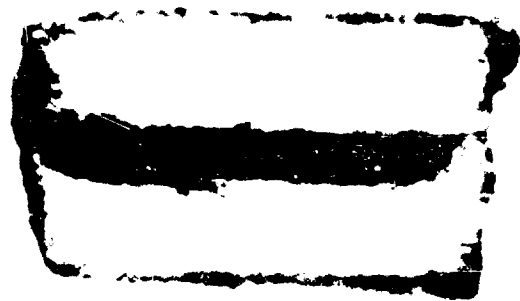
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1. *Thymus praecox* and the cooling of 10° for 1 minute.
2. *Thymus praecox* and the cooling of room temperature for 1 minute.
3. *Thymus praecox* and the cooling of 10° for 1 minute.
4. *Thymus praecox* and the cooling of 10° for 1 minute.
5. *Thymus praecox* and the cooling of 10° for 1 minute.
6. *Thymus praecox* and the cooling of 10° for 1 minute.
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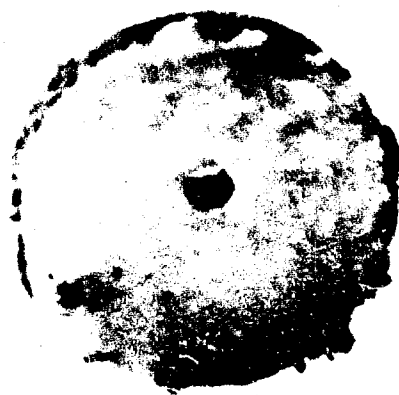
September 1953

PICATINNY ARSENAL

ORDNANCE CO-175

M-17 Cal. .50 Propellant coated with 2-nitrodiphenylamine (a) Immediately after coating (b) After 6 hours (c) After 24 hours

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M-17 Cal. .50 Propellant coated with Copper Resinate/
Dicyclohexylphthalate: 70/30

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